

Fatigue and Damage Tolerance of Y-TZP Ceramics in Layered Biomechanical Systems

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Abstract: The fatigue properties of fine-grain Y-TZP in cyclic flexural testing are studied. Comparative tests on a coarser-grain alumina provide a baseline control. A bilayer configuration with ceramic plates bonded to a compliant polymeric substrate and loaded with concentrated forces at the top surfaces, simulating basic layer structures in dental crowns and hip replacement prostheses, is used as a basic test specimen. Critical times to initiate radial crack failure at the ceramic undersurfaces at prescribed maximum surface loads are measured for Y-TZP with as-polished surfaces, mechanically predamaged undersurfaces, and after a thermal aging treatment. No differences in critical failure conditions are observed between monotonic and cyclic loading on as-polished surfaces, or between as-polished and mechanically damaged surfaces in monotonic loading, consistent with fatigue controlled by slow crack growth. However, the data for mechanically damaged and aged specimens show substantial declines in sustainable stresses and times to failure in cyclic loading, indicating an augmenting role of mechanical and thermal processes in certain instances. In all cases, however, the sustainable stresses in the Y-TZP remain higher than that of the alumina, suggesting that with proper measures to avoid inherent structural instabilities, Y-TZP could provide superior performance in biomechanical applications. © 2004 Wiley Periodicals, Inc. *J Biomed Mater Res Part B: Appl Biomater* 71B: 166–171, 2004

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INTRODUCTION

Ceramic-based biomechanical components in dental crowns and total hip replacement (THR) prostheses often take the form of brittle ceramic layers on compliant polymeric support sublayers.^{1–5} The hard ceramic layers protect the soft underlayers by sustaining the bulk of the operational stresses. The underlayers in turn provide energy absorption and toughness. Alumina has long been the benchmark ceramic for such applications because of its chemical and thermal stability, but its continued use has been questioned because of its brittleness—its fracture strength is only moderate (~400–600 MPa). Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) has been suggested as a replacement material—it has substantially higher strength (>1 GPa) as well as slightly

higher toughness.⁶ However, Y-TZP is less stable hydrothermally.³ Consequently, questions remain concerning the longevity of Y-TZP in the human body, especially under the repetitive stress concentrations in environments that typify prosthetic function.

There has been a large volume of literature concerning the fatigue properties of Y-TZP. Several authors have reported fatigue from some combination of slow crack growth and mechanical degradation in traditional fracture specimens containing artificially introduced long cracks.^{7–10} However, fatigue responses in the long-crack region can differ greatly from those in the short-crack region that typifies well-finished components.¹¹ Such a finished component may be highly surface polished, so that the laboratory strength is determined by intrinsic flaws within the microstructure. Tension–compression strength tests on well-polished Y-TZP rods^{12,13} have indicated cyclic fatigue associated with microcrack flaws, with some conjecture as to the origin of mechanical degradation at reverse-sliding microcrack faces.^{12,14} At the same time, little attention has been paid to lifetime properties in terms of extrinsic-flaw states—the same initially well-polished component may accumulate substantial damage during fabrication and finishing, heat sterilization (aging), and even

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surgery and *in vivo* function. The question arises: How susceptible is Y-TZP to degradation associated with short-crack behavior associated with small-scale intrinsic and extrinsic flaw states?

The current study examines the fatigue responses of Y-TZP in repetitive flexural stressing. The tests are carried out with the use of a bilayer configuration in which the ceramic plates are first bonded to a polycarbonate substrate base and then loaded to failure at the top surface with a spherical indenter, with the substrate on a flat support.^{15–17} The contact loading causes the ceramic plate to flex on its compliant support, placing the bonded lower surface in biaxial tension. Strengths are calculated from the critical loads to cause failure in the plate. The test simulates the basic layer elements of a range of ceramic-based crown/dentin and liner/UHMWPE acetabular cup or femoral-head/acetabular THR structures.^{5,17} Fatigue testing is carried out by applying the load in a single half-cycle at a fixed rate (dynamic fatigue) or sinusoidally (cyclic fatigue). Comparison of data from these tests enables the contributions to fatigue from slow crack growth and mechanical sources to be differentiated. Initial tests are conducted on well-polished Y-TZP surfaces. Analogous tests on polished alumina plates are conducted to provide a reference baseline. Comparative tests are run on Y-TZP specimens after introducing controlled predamage into the tensile undersurfaces, and after preaging treatments. It is concluded that Y-TZP can be a resilient material over extended lifetimes, at least over a wide range of normal handling and aging conditions.

MATERIALS AND METHODS

Materials and Testing

Dense medical grade 3 mol % yttria-stabilized zirconia (Prozr Y-TZP, Norton, East Granby, CT) and alumina (AD995, CoorsTek, Golden, CO) were used as test ceramics. Specimens were ground and polished from supplied stock as plates measuring $25 \times 25 \times 0.6$ mm (Y-TZP) and $25 \times 25 \times 1$ mm (alumina). X-ray diffraction indicated no transformed monoclinic phase at the polished surfaces (within $\sim 3\%$ detection limit). Microstructures of these materials are shown in Figure 1. Note the relatively fine (submicrometer), homogeneous and equiaxed structure of Y-TZP. An epoxy resin (Harcos Chemicals, Bellesville, NJ) was used to bond the ceramic plates to clear polycarbonate substrate blocks 12.5 mm thick (Hyzod, AIN Plastics, Norfolk, VA) to form a bilayer configuration. The procedure used to fabricate the ceramic/polycarbonate bilayers has been well documented in previous studies.^{15,16,18} The epoxy bonding interlayer between ceramic and substrate is typically ~ 10 μm , and is clear. The interlayer thickness is not an important factor in the present study, because the elastic modulus of epoxy resin is similar to that of the polycarbonate base.¹⁸

The ceramic/polycarbonate bilayers were loaded at their top surfaces with tungsten carbide (WC) spheres of radius

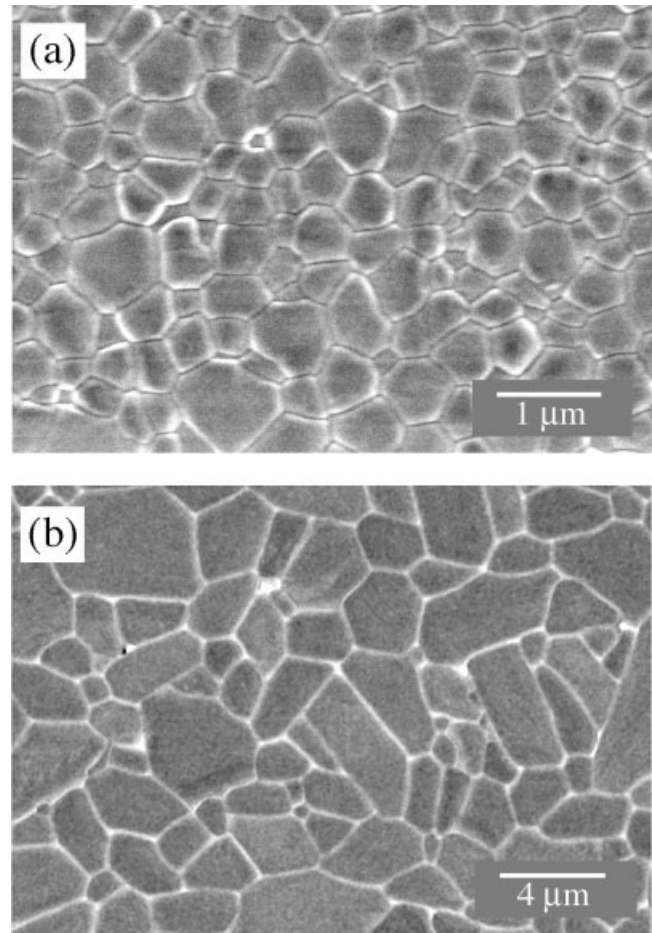


Figure 1. Microstructures of yttria-stabilized zirconia (Y-TZP) and alumina (Al_2O_3). Thermally etched surfaces, SEM.

3.18 mm mounted into the cross heads of mechanical testing machines (Figure 2). Loading was applied in two modes: (a) constant monotonically increasing rates (dynamic fatigue), on a screw-driven testing machine (Model 5500R, Instron, Corp, Canton, MA); and (b) cyclic loading (cyclic fatigue), between near-zero and maximum load at frequencies 0.1 and 10 Hz, on a hydraulic testing machine (Model 8500, Instron Corp., Canton, MA). The ceramic undersurfaces were monitored from below the contact through the transparent adhesive/polycarbonate substrate by a video camcorder (Canon XL1, Canon, Lake Success, NJ) equipped with a microscope zoom system (Optem, Santa, VA). Critical loads to radial crack failures at the ceramic undersurfaces were thereby measured directly, and the corresponding test durations recorded.

The bulk of the tests were conducted on ceramics with as-polished surfaces. Other tests were carried out after subjecting Y-TZP plates to some form of degradation treatment:

1. Controlled surface damage: Polished ceramic undersurfaces were pre-damaged at their centers by indenting with a WC sphere of radius 1.98 mm to loads 3000 and 4000 N, as an instance of severe (controlled) handling damage.¹⁹ The contact damage has the form of a quasiplasticity zone

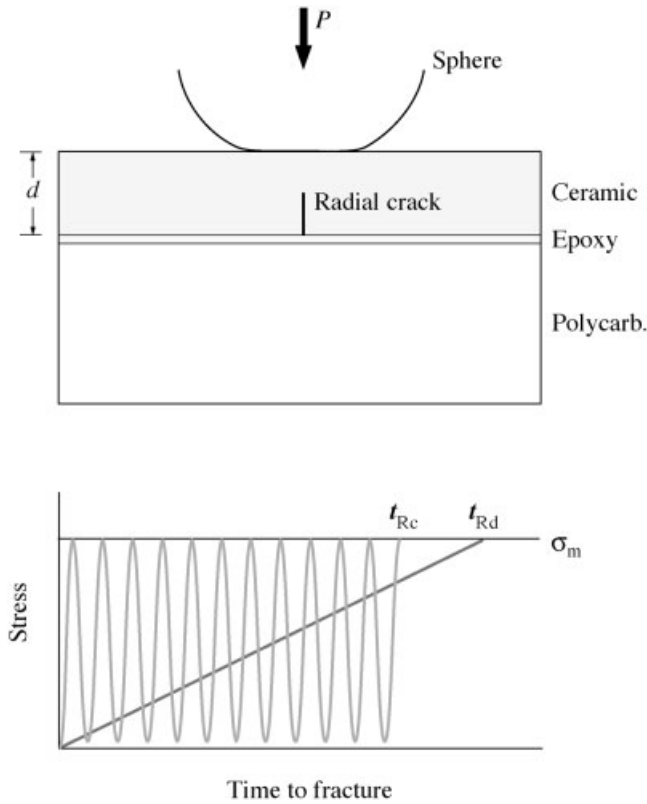


Figure 2. Schematic of bilayer test specimen, ceramic plate of thickness d and modulus E_c bonded to polycarbonate substrate of modulus E_s . Loading with sphere at top surface produces radial crack at undersurface. Loading is performed at constant stress rate (dynamic fatigue) or sinusoidally (cyclic fatigue).

with incipient microcracks—the chosen loads embrace critical conditions to cause microcrack coalescence within this zone.¹⁹ Surface and subsurface damage was examined by a bonded-interface sectioning technique.²⁰

2. Heat treatment: Y-TZP plates were rapid heated in a furnace (Omegalux™ LMF-6525, Omega Engineering Inc., Stamford, CT) to 200°C and aged for 200 h, in air. Relative amounts of tetragonal (t) and monoclinic (m) phases before and after the treatment were measured by X-ray diffractometry (XRD) (D500, Siemens Corp.). Surfaces were examined using Nomarski contrast microscopy.

Fracture Mechanics

Appropriate fracture mechanics for the test configuration in Figure 2 have been described elsewhere,¹⁶ and only the essential relations will be presented here. The concentrated load P at the ceramic plate top surface induces a maximum flexural tensile stress at the center of the plate undersurface^{18,21}

$$\sigma = (P/Bd^2)\log(E_c/E_s) \quad (1)$$

where d is the plate thickness, E_c and E_s are Young's modulus of ceramic plate and polymer substrate, respectively, and B =

1.35 is a dimensionless coefficient.^{22,23} Radial cracking initiates from a dominant starting flaw in the ceramic at or close to the central location of this maximum tensile stress. The starting flaw is subject to moisture-assisted slow crack growth, expressed by a crack velocity relation $v \sim K^N$,²⁴ where N is a characteristic exponent and $K \sim \sigma c^{1/2}$ is a stress-intensity factor for a crack of length c under tensile stress σ .²⁵ Combining these basic relations with some prescribed time-dependent loading function $P(t)$ and integrating over the time t_R to grow the initial flaw to instability then yields critical conditions for radial fracture.

The two types of loading of interest here are:

1. Constant stressing rate (dynamic fatigue): The specimen is loaded at fixed rate $dP/dt = \text{constant}$ to failure at critical stress σ_m . The failure condition is¹⁶

$$\sigma_m^N t_{Rd} = A(N+1) \quad (2)$$

where A is a load-, time- and thickness-independent quantity and subscript d denotes dynamic loading.

2. Constant frequency (cyclic fatigue): The specimen is loaded sinusoidally between zero and maximum σ_m at a constant frequency f . The failure condition is¹⁶

$$\sigma_m^N t_{Rc} = 2AN^{0.47} \quad (3)$$

where subscript c denotes cyclic loading.

If slow crack growth is the sole contributing factor in fatigue, then fatigue data for any given flaw state can be reduced to an equivalent cyclic function $\sigma_m(t_R)$ with effective fracture times (Figure 2):

$$t_R = t_{Rc} \quad (\text{cyclic test}) \quad (4a)$$

$$t_R = [2N^{0.47}/(N+1)]t_{Rd} \quad (\text{dynamic test}) \quad (4b)$$

the latter equation obtained by dividing Equation (2) into Equation (3).

RESULTS

Figure 3 shows stress–time results for the Y-TZP and alumina bilayers, for both dynamic and cyclic tests. This figure is plotted as follows:

1. Calculate maximum stresses $\sigma = \sigma_m$ from critical loads $P = P_R$ for radial cracking in Equation (1), with modulus $E_c = 205$ GPa for Y-TZP and $E_c = 372$ GPa for alumina ceramics, $E_s = 2.3$ GPa for polycarbonate substrate.²⁶
2. Regression best-fit the raw dynamic fatigue $\sigma_m(t_{Rd})$ data in accordance with the power-law relation in Equation (2)

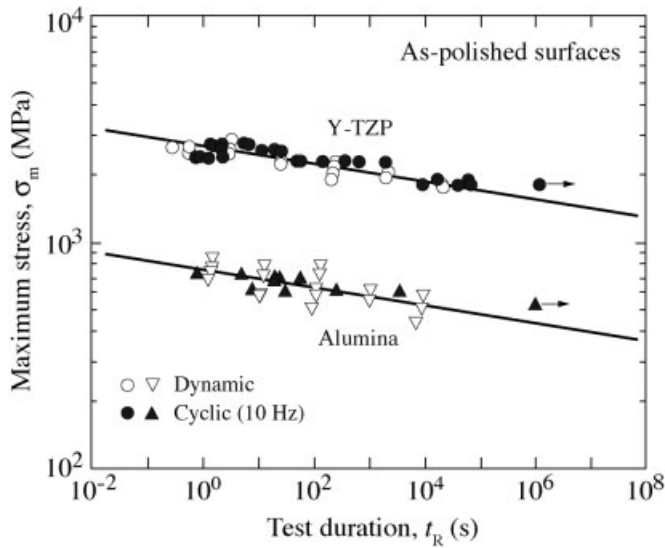


Figure 3. Maximum applied tensile stress σ_m as function of effective time to fracture t_R for as-polished plates of thickness 0.6 mm (Y-TZP) and 1.0 mm (alumina) in top-loaded bilayer test specimens (Figure 2). Failure occurs by initiation of radial cracks from flaws at the ceramic undersurface. Data represent individual tests at constant monotonic stressing rates (unfilled symbols) and in cyclic loading at 10 Hz (filled symbols). Solid lines are data fits in accordance with slow crack growth relations. Arrows indicate runouts.

to determine $N = 25 \pm 2$ for Y-TZP and $N = 26 \pm 6$ for alumina, as well as A for each material.

3. Plot individual $\sigma_m(t_R)$ dynamic and cyclic data for each material with the use of Equation (4) to determine effective fracture times (arrows indicate runouts).
4. Plot solid lines corresponding to predictions for cyclic fatigue functions $\sigma_m(t_{Rc})$ with the best-fit values of N and A in Equation (3) for each material. The cyclic and dynamic fatigue data in this plot overlap each other over the data range, within the experimental scatter, consistent with the slow crack growth model. However, the sustainable stress levels in the zirconia are substantially higher than in the alumina, by a factor > 3 at any prescribed test duration.

Figure 4 shows analogous $\sigma_m(t_R)$ data for Y-TZP specimens after predamage with 3000 N indentations from a 1.98 mm radius sphere. Data are for tests in cyclic fatigue (filled symbols) at frequencies 0.1 and 10 Hz, and for some limited tests in dynamic fatigue (unfilled symbols). The solid line is the data fit for as-polished Y-TZP surfaces in Figure 3; the dashed line is the corresponding as-polished data fit for alumina, included as a reference baseline. In all cases, failure initiated preferentially through the contact damage site. This is not surprising, because the indentations measure about 500 μm in surface diameter, and thus occupy the central region of maximum tensile stress at the ceramic undersurface (concentrated in area $\sim d^2 = 1 \text{ mm}^2$).²² The dynamic data show no shifts in lifetime within the experimental scatter, suggesting that the strength-degrading influence of the 3000 N contacts

is miniscule in monotonic loading. However, after an initial falloff within $t_R \sim 1\text{--}100 \text{ s}$, the cyclic fatigue data show substantial shifts to lower sustainable stresses or smaller lifetimes, tending closely parallel to the dynamic data in the long-lifetime region. The data at 10 Hz are particularly strongly shifted in this latter region, amounting to a reduction $\sim 30\%$ in sustainable stress. Note, however, that even these diminished stresses remain well above those for as-polished, unindented alumina, by well over a factor of 2.

Figure 5 compares results of stress to failure for Y-TZP specimens in the as-polished state and after indentation pre-damage at $P = 3000$ and 4000 N , at a common constant stressing rate 10 MPa s^{-1} . Consistent with Figure 4, the data indicate virtually no decrease in strength resulting from the 3000 N indentations, relative to the as-polished surface state. However, the strengths of specimens with 4000 N indentations indicate a relative falloff of $\sim 50\%$. These results are consistent with previously reported trends for indentation-damaged Y-TZP.¹⁹ Surface views of the indentation sites revealed some tendency to shallow ring cracking at 4000 N, but not at 3000 N. Interestingly, the specimens continued to break through rather than around the contact damage sites, suggesting that microcrack coalescence must have occurred within the quasiplastic damage zone interior at the higher indentation load.²⁷

Figure 6 illustrates the effect of aging at 200°C for 200 h, in air, on the $\sigma_m(t_R)$ response for polished Y-TZP. The data are for cyclic tests at 10 Hz. Again, the lines represent the data fits for as-polished Y-TZP and alumina from Figure 3. In this case the decrease in sustainable stresses relative to as-polished surfaces is $\sim 40\%$, but still a factor of 2 higher than

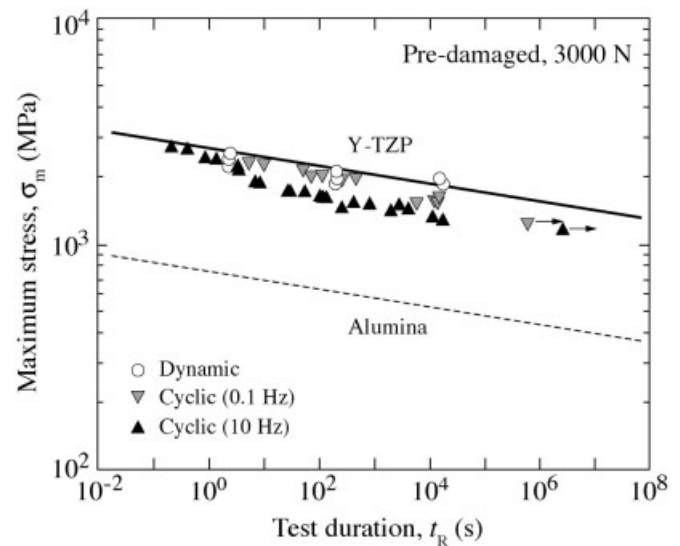


Figure 4. Similar $\sigma_m(t_R)$ plot to Figure 3, but for Y-TZP after predamage at the ceramic undersurface with 3000 N indentations with a 1.98 mm radius WC sphere. Data for tests in cyclic loading (filled symbols) at frequencies 0.1 and 10 Hz, and at constant stressing rates (unfilled symbols). Solid and dashed lines are fits for as-polished Y-TZP and alumina surfaces from Figure 3. Failure initiated preferentially through the contact predamage sites in all cases. Arrows indicate runouts.

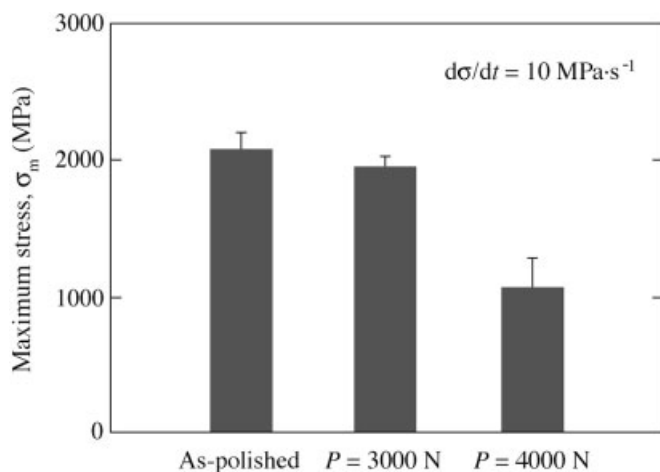


Figure 5. Bar diagram comparing stresses σ_m to failure in Y-TZP at a fixed monotonic stressing rate 10 MPa s^{-1} for as-polished and pre-damaged surfaces at indentation loads $P = 3000$ and 4000 N.

as-polished alumina. XRD analysis of the Y-TZP before and after aging indicated an increase in m -phase content from zero to ~ 70 vol %. Surface examinations in Nomarski contrast showed substantial surface rumpling as a result of this transformation.

DISCUSSION

In this study dynamic (constant stressing rate) and cyclic loading experiments have been used to evaluate the fatigue properties of Y-TZP, with alumina as a reference control, in air. A simple bilayer test configuration in which the test ceramic is bonded to a polycarbonate base and loaded at its upper surface in Hertzian contact has been used to simulate some of the more basic elements of crown and THR prosthetic layer structures. This configuration places the ceramic overlayer in flexural loading, in analogy to a conventional biaxial bend test, with resultant failure by initiation of radial cracks from flaws at the lower ceramic surface. For as-polished surfaces (Figure 3), the Y-TZP outperforms the alumina, requiring some threefold higher applied stresses to cause failure at an equivalent loading time. This higher stress level correlates directly with the comparatively high strength of Y-TZP, attributable to its fine grain size (Figure 1) and (less importantly) to a slightly higher toughness, relative to alumina. Again for as-polished surfaces, there is no measurable difference in fatigue data obtained from dynamic and cyclic loading, consistent with a dominant role of slow crack growth in the fatigue process under these conditions. The slow crack growth is associated with access of moisture to the ceramic undersurface from water content in the adhesive/substrate support materials.¹⁶ The results in Figure 3 suggest that simple constant loading rate testing on specimens with polished ceramic surfaces can be used to obtain critical fatigue data for lifetime predictions of well-finished components.²⁶

However, questions arise as to whether Y-TZP can sustain its superior performance when exposed to potentially severe extraneous degradation conditions, either in fabrication and preparation or during service. One of these conditions relates to mechanical damage from spurious surface contacts or impacts, as might be encountered in prosthesis handling and insertion procedures. Potential damage of this kind has been investigated here by artificially introducing quasiplastic damage into the ceramic tensile undersurfaces by sphere indenters (radius 1.98 mm) prior to bonding to the polycarbonate substrate.²² Figure 4 shows that whereas indentations at 3000 N do not degrade the ensuing strength properties in monotonic loading, the same indentations cause substantial loss of strength in cyclic loading, especially at higher frequencies. This enhancement of strength loss in cyclic loading indicates augmentation of slow crack growth by mechanical degradation processes. The quasiplastic zone in Y-TZP contains incipient microcracks, in themselves not immediately deleterious but highly capable of coalescing into strength-degrading macroscale fractures under repeat high-stress loading.¹⁹

A second kind of degradation condition in Y-TZP relates to material aging that might be encountered in sterilization or other preparation treatments. Thus the data in Figure 6 reveal substantial degradation from heat treatments in air at 200°C for 200 h . Such aging processes are known to weaken the material by inducing t - m transformations, with attendant microcracking.²⁸ As indicated, the degree of transformation associated with the heat-treatment procedure represented in Figure 6 is ~ 70 vol%. This is considerably higher than the ~ 20 – 30% reported for retrieved Y-TZP femoral heads in THR revision case studies,²⁹ suggesting that the current test conditions are at least as severe as those experienced *in vivo*. At the same time, the transformation process induces sub-

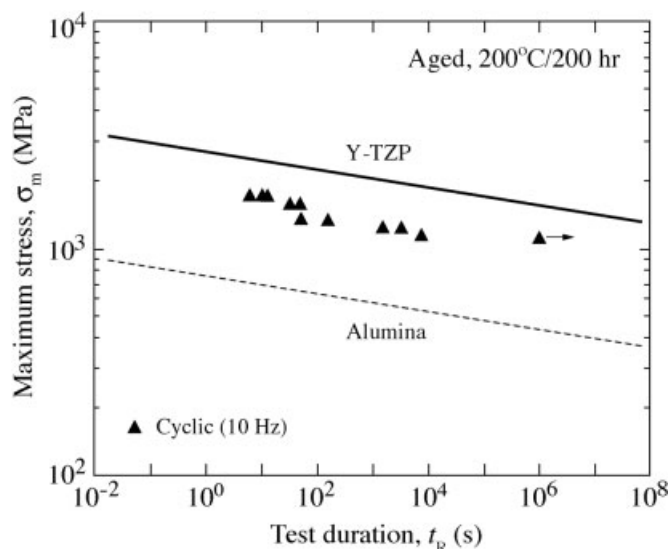


Figure 6. Similar $\sigma_m(t_R)$ plot to Figure 4, but for Y-TZP after aging at 200°C for 200 h , in air. Initially polished surfaces show rumpling after heat treatment. Data are for cyclic loading at 10 Hz . Solid and dashed lines are fits for as-polished Y-TZP and alumina surfaces from Figure 3. Arrow indicates runouts.

stantial surface rumpling, which can be highly deleterious to wear properties at moving femoral-head/acetabular-cup contact surfaces.⁶

Taken together, the results in Figures 4–6 may be viewed as confirmation of some mechanical and thermodynamic instability in the Y-TZP structure. The inherent instability of Y-TZP remains an issue that demands continued attention. Strict control of processing procedures is essential to ensure that the material is microstructurally stable during fabrication. Care must also be taken to ensure that the material does not experience any thermal, mechanical, or chemical microstructure-altering treatments during its subsequent operational lifetime.^{3,6,30} Relative to other zirconias, Y-TZP is brittle and especially susceptible to fracture from sharp-contact damage, so scratching from particulates must also be avoided.^{31,32} Nevertheless, in all tests Y-TZP remained stronger than as-polished alumina, consistent with its relatively small grain size. On the face of it, therefore, the present results might be taken as justification for Y-TZP as useful replacement material for alumina in biomechanical prostheses, provided it can be protected from inadvertent, excessive degradation. It may well serve, for example, as core materials in dental crowns, where the surfaces are remote from moving surfaces and therefore somewhat protected from external influences. The search for alternative ultra-strong but stable material systems, for example, matrix-reinforced aluminas,³ would appear to be a wise course for future research.

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